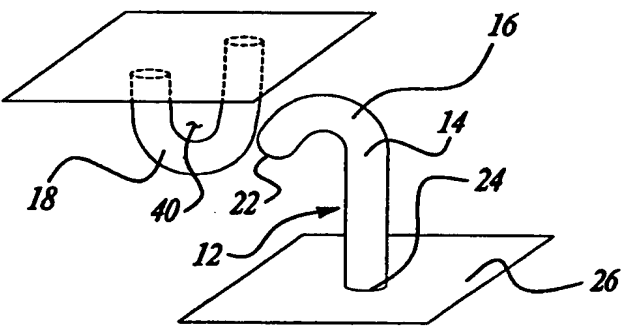


INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification ⁶ : A44B 18/00	A1	(11) International Publication Number: WO 99/40812 (43) International Publication Date: 19 August 1999 (19.08.99)
(21) International Application Number: PCT/US99/02897 (22) International Filing Date: 11 February 1999 (11.02.99) (30) Priority Data: 60/074,463 12 February 1998 (12.02.98) US (63) Related by Continuation (CON) or Continuation-in-Part (CIP) to Earlier Application US 60/074,463 (CON) Filed on 12 February 1998 (12.02.98) (71) Applicant (for all designated States except US): BOARD OF TRUSTEES OPERATING MICHIGAN STATE UNIVERSITY - [US/US]; East Lansing, MI 48824 (US). (72) Inventors; and (75) Inventors/Applicants (for US only): TOMANEK, David [US/US]; 1136 Poplar Lane #4, East Lansing, MI 48823 (US). ENBODY, Richard [US/US]; 345 University Drive, East Lansing, MI 48823 (US). KWON, Young-Kyun [KR/US]; 1443D Spartan Village, East Lansing, MI 48823 (US).		(74) Agents: SIMINSKI, Robert, M. et al.; Harness, Dickey & Pierce, P.L.C., P.O. Box 828, Bloomfield Hills, MI 48303 (US). (81) Designated States: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, GH, GM, HR, HU, ID, IL, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG). Published <i>With international search report.</i> <i>Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.</i>
(54) Title: MICRO-FASTENING SYSTEM AND METHOD OF MANUFACTURE		
(57) Abstract		
<p>The present invention relates to a micro-fastening system and, more particularly, to a mechanical micro-fastening system employing a plurality of mating nanoscale fastening elements (16, 18) and a method of manufacturing a micro-fastening system in accordance with the teachings of the present invention. The mating nanoscale fastening elements (16, 18) are formed by functionalizing nanotubes having an ordered array of hexagons with pentagons and heptagons at particular heterojunctions.</p> 		

FOR THE PURPOSES OF INFORMATION ONLY

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AL	Albania	ES	Spain	LS	Lesotho	SI	Slovenia
AM	Armenia	FI	Finland	LT	Lithuania	SK	Slovakia
AT	Austria	FR	France	LU	Luxembourg	SN	Senegal
AU	Australia	GA	Gabon	LV	Latvia	SZ	Swaziland
AZ	Azerbaijan	GB	United Kingdom	MC	Monaco	TD	Chad
BA	Bosnia and Herzegovina	GE	Georgia	MD	Republic of Moldova	TG	Togo
BB	Barbados	GH	Ghana	MG	Madagascar	TJ	Tajikistan
BE	Belgium	GN	Guinea	MK	The former Yugoslav Republic of Macedonia	TM	Turkmenistan
BF	Burkina Faso	GR	Greece			TR	Turkey
BG	Bulgaria	HU	Hungary	ML	Mali	TT	Trinidad and Tobago
BJ	Benin	IE	Ireland	MN	Mongolia	UA	Ukraine
BR	Brazil	IL	Israel	MR	Mauritania	UG	Uganda
BY	Belarus	IS	Iceland	MW	Malawi	US	United States of America
CA	Canada	IT	Italy	MX	Mexico	UZ	Uzbekistan
CF	Central African Republic	JP	Japan	NE	Niger	VN	Viet Nam
CG	Congo	KE	Kenya	NL	Netherlands	YU	Yugoslavia
CH	Switzerland	KG	Kyrgyzstan	NO	Norway	ZW	Zimbabwe
CI	Côte d'Ivoire	KP	Democratic People's Republic of Korea	NZ	New Zealand		
CM	Cameroon			PL	Poland		
CN	China	KR	Republic of Korea	PT	Portugal		
CU	Cuba	KZ	Kazakstan	RO	Romania		
CZ	Czech Republic	LC	Saint Lucia	RU	Russian Federation		
DE	Germany	LI	Liechtenstein	SD	Sudan		
DK	Denmark	LK	Sri Lanka	SE	Sweden		
EE	Estonia	LR	Liberia	SG	Singapore		

TITLE

MICRO-FASTENING SYSTEM AND METHOD OF MANUFACTURE

BACKGROUND OF THE INVENTION

1. Field of the Invention

The present invention relates to a micro-fastening system and, more particularly, to a mechanical micro-fastening system employing a plurality of mating nanoscale fastening elements and a method of manufacturing the same.

2. Description of the Prior Art

Micro-fastening systems per se are utilized to connect distinct components brought into relative contact by strong bonds which span a gap at the interface and generally are less than one micrometer in size. In their most common embodiments, such microfastening systems have generally been in the form of chemical bonds such as adhesive bonds, welds and coatings. Numerous potential disadvantages associated with employing adhesives and coatings are known such as the irreversible nature of the bonds and the potential for degradation at relatively high temperatures. Further, adhesives and coatings generally require smooth dry interfaces which are free of impurities to effectuate high quality bonds. Welding results in a physical deformation of the surfaces being welded; it cannot be used effectively for interconnecting microscopically small components or large interface areas. Thus, there is a need for the mechanical "micro-fastening" system of the present invention.

SUMMARY OF THE INVENTION

The micro-fastening system of the present invention employs a plurality of mating nanoscale fastening elements which are obtained by structurally modifying, i.e., functionalizing nanotubes generally and carbon nanotubes particularly. Carbon nanotubes per se consist of a graphite monolayer having the overall shape of a cylinder including an ordered array of hexagonal carbon rings disposed along the cylindrical side walls which may be single or

multi-walled as reported in *Nature*, Vol. 354 (1991) pp. 56 - 58 and *ibid.* Vol. 363 (1993) pp. 603 - 605. The ends of the tubes are often closed by pairs of pentagonal carbon rings. Carbon nanotubes generally range in diameter from one to about 50 nanometers, and may be as long as approximately 0.1 millimeters. While related to carbon fibers, nanotubes are free of atomic scale defects, which accounts for their high tensile strength, as compared to that of the strength of individual graphite layers. Like graphite, carbon nanotubes exhibit sp^2 bonding which gives rise to a relatively high degree of flexibility and resilience. Further, carbon nanotubes are structurally stable nearly up to the melting point of graphite, i.e., up to about 3,500 degrees Celsius.

By functionalizing the carbon nanotubes as will be described in greater detail below, the cylindrical shape can be modified to include bent portions. While it has been suggested generally that carbon nanotubes can be readily functionalized, it has yet to be reported that carbon nanotubes can be specifically functionalized so as to obtain mating fastening elements as herein described.

Among the various applications for the micro-fastening system of the present invention are the assembly of nano-robots useful for micro-surgical procedures, surface coatings, and attachment of metal contacts to integrated semiconductor devices, by way of non-limiting example.

The strength of micro-fastening systems described herein relies on the enormous stability of nanotubes, i.e., their large structural rigidity, the high strength of the bonds anchoring tubes in a substrate and a large number of connections possible on a limited surface area. In contrast to purely mechanical fasteners (such as bolts and screws) which weaken the surfaces to be connected, there is no apparent degradation of the opposing surfaces to be joined under the present invention. Adhesives are typically weaker than most mechanical fasteners and their strength is strongly diminished at higher temperatures. Welding is not practicable for large interfaces, whereas the fastening system of the present invention may be employed for both large and microscopically small interfaces. Bonding technologies excepting the

micro-fastening system of the present invention leave macroscopically large gaps at the interface. Unlike known bonds between substrates, the micro-fastening system of the present invention has an effective thickness of the gap at interface as small as a few nanometers.

5 A further advantage of the present invention is that the surface bonds based on the nanotube based micro-fastening system, while extremely strong, may be re-opened and re-closed, i.e., they are reusable, whereas the surface bonds generated by gluing or welding are permanent. Thus, the micro-fastening system of the present invention is selectively reversible which is
10 considered to be highly desirable, particularly for self-repair. This reusability or self-repairability is of particular advantage for interconnects exposed to changing forces or changing environmental variables (such as temperature) that result in a different expansion of the individual components brought into relative contact.

15 Still another advantage offered by the micro-fastening system of the present invention is that the conductivity of the fastening elements connecting the corresponding substrates may be varied from metallic to insulating, depending largely on the chemical composition, the diameter and chirality of the nanotubes.

20 **BRIEF DESCRIPTION OF THE DRAWINGS**

Figures 1(a-c) are a series of views demonstrating the representative closure mechanism and forces for a generic micro-fastening system in accordance with the teachings of the present invention.

25 Figures 1(d-f) are a series of views demonstrating the representative opening mechanism and forces for a micro-fastening system in accordance with the teachings of the present invention.

Figure 2 is a schematic view illustrating a way to define the figure of merit of the micro-fastening system wherein the horizontal axis X represents
30 the separation between the surfaces.

Figures 3(a-d) are a series of views demonstrating the representative opening and closure mechanisms and forces for a particular micro-fastening

system based on nanotubes functionalized to form a mating hook and loop arrangement in accordance with the teachings of the present invention.

Figures 4(a-b) are illustrative of alternative mating nanoscale micro-fastening system elements in accordance with the teachings of the present invention.

DETAILED DESCRIPTION OF THE INVENTION

The micro-fastening system 10 of the present invention comprises a plurality of mating nanoscale fastening elements 12 and 12' manufactured by modifying, i.e., functionalizing nanotubes which are generally linear in nature prior to functionalizing. Upon functionalizing the nanotubes 14, fastening elements are obtained in a variety of non-linear forms such as hooks 16 and loops 18 as illustrated in Figs. 3(a-d) and spirals 20 as illustrated in Fig. 4(b) by way of non-limiting example. The nanotubes employed may be composed of carbon, nitrogen, boron or other elements which give rise to layered honeycomb lattice structures. It is important from the outset to note that the nanotubes employed in accordance with the teachings of the present invention may be single walled, multi-walled or at least partially multi-walled over the length of the nanotube. For simplicity, the present invention will hereinafter generally be described in terms of functionalizing graphitic carbon nanotubes.

By "functionalizing" graphitic carbon nanotubes, it is meant that a specific number of pentagons and heptagons are substituted for hexagons within the nanotube or are added along the open edge(s) of the core nanotube which consists of an ordered array of hexagons.

Upon introducing pentagons and heptagons in a predetermined order, the carbon nanotubes will exhibit a locally positive or negative Gaussian curvature that results in a "bend" in the nanotube. By continuing to add pentagons and hexagons in a specific manner, the bend of the nanotube can be grown until the desired shape is obtained.

Upon growing the carbon nanotube to the desired length and shape, a first end 22 of the nanotube 14 may be capped or terminated, e.g., by

introducing or forming a fullerene half dome along the end to be terminated. By providing a fullerene half-dome along an open end of the carbon nanotube, the end of the formed fastening element 12 becomes substantially inert, i.e., non-bonding to other atoms or molecules.

5 A second end 24 of the fastening element which is open, i.e., non-terminated, is bonded to a substrate 26 which may be in the form of various materials including metals, carbon (graphite or diamond), silicon, germanium, polymers and composites of the foregoing, to name a few. Other materials, provided they are capable of attaining a molten state, can also be employed.

10 Since the open end 24 of the nanotube is highly reactive and thus has a natural affinity for bonding to the desired substrate, the fastening element readily attaches to the substrate in a manner whereby the element stands up along the attachment surface. Nanotubes may be assisted in their alignment perpendicular to the surface by applying a strong electric field in that direction. This so-called affinity to migrate toward the surface is at least
15 partially due to the low surface tension of the nanotube material. As will be understood by those skilled in the art, the tendency for the fastening elements to stand up promulgates mating between corresponding fastening elements.

Carbon nanotubes having ordered pairs of pentagons and heptagons
20 may occur spontaneously to a limited extent during synthesis, thus forming hook shaped nanotubes as reported in MRS Bulletin, Vol. 19, No. 11, pp 43 - 49 (1994). However, in order to design carbon nanotubes such that they can be used effectively in micro-fastening systems, atomically dispersed catalysts may be necessary. For example, transition metals such as Fe and, more
25 preferably, Ni, Co and Y have been shown to promote formation of single wall nanotubes or spiral structures as reported in Science 265, 635 (1994).

Curvature of the ends or other portions of relatively straight carbon nanotubes can be also accomplished by employing a template in proximity to a growing nanotube. In this regard, both on energetic and entropic
30 grounds, a horizontally growing nanotube, when approaching a vertically positioned nanotube used as a template, has a higher probability to form ordered pairs of C_5 and C_7 carbon rings, i.e., pentagons and heptagons which

would cause the former to "wrap around" the latter. As such, specifically functionalized carbon nanotubes 14 useful as fastening elements 12 such as those illustrated in Figs. 4(a-b) can also be prepared without employing catalysts.

5 As shown in Figs. 1(a-c), only a moderate force F_c is required to selectively deform the nanotube and thereby accomplish an interconnection between the first and second fastening elements 12 and 12'. A much larger force F_o is required to break the interconnection between the fastening elements 12 and 12' of components in contact as demonstrated in Figs. 1(d-
10 f). The hatched area in Fig. 2 represents the work required to close and re-open the gap and indicates the efficiency of a particular pair of mating nanoscale fastening elements.

As noted, while the fastening elements 12 and 12' can be formed into a number of different configurations, certain configurations are considered to
15 be preferred. For a generic mechanical micro-fastening system, the opening and closing mechanism is shown in Figs. 1(a-f). Generic fastening elements, shown in these figures, contain a substantially triangular shaped head 30. Under this schematic embodiment the angled surfaces 32 and 32' slide past the other as the fastening elements come into contact as they advance
20 toward an interlocked position. This angular orientation of approximately 45° along surfaces 32 and 32' allows for a minimal amount of lateral deflection of the fastening elements during the attachment step. The attachment surfaces 34 and 34' preferably slope downwardly and away from their respective stems 36 and 36' to form an interconnection requiring a relatively high separation
25 force, i.e., $|F_o| > |F_c|$.

Figs. 3(a-d) show one particular embodiment of the micro-fastening system, consisting of hook 16 and loop 18 fastening elements. Under this embodiment, as the hook and loop elements are advanced toward each other, the first end 22 of the hook deflects until there is sufficient clearance
30 to insert into the aperture 40 of the loop element. As with the embodiment illustrated in Figs. 1(a-f), the hook and loop fastening system requires a

relatively high separation force $|F_o| > |F_c|$ to detach the fastening elements as compared to the attachment forces.

5 Still other embodiments such as hook 16 to hook 16' fastening as illustrated with reference to Fig. 4a and spiral 20 to hook 16 fastening as illustrated in Fig. 4b are considered as practical applications. In essence, the shape of the resulting fastening elements is a function of the processing parameters, as such various fastening element configurations are contemplated.

10 Additionally, it should be understood that micro-fastening elements having different shapes can be formed upon the same substrate. Thus, alternating rows of specifically shaped fastening elements along a useful substrate is an effective application. Of course, microfastening elements of differing configurations can be randomly applied to a substrate, if desired.

15 While it will be apparent that the preferred embodiments of the invention disclosed are well calculated to fulfill the objects stated, it will be appreciated that the invention is susceptible to modification, variation and change without departing from the spirit thereof.

CLAIMS

1. A microfastening system comprising:
a first fastening element including a plurality of extending
nanotubes; and

5 a second fastening element including a plurality of extending
nanotubes;

whereby upon joining said first and second fastening elements,
the extending nanotubes from each element become mechanically
interconnected.

10 2. The microfastening system of Claim 1 wherein said at least one
of first and second fastening elements further comprise a substrate from
which said nanotubes extend.

15 3. The microfastening system of Claim 2 wherein said substrate is
formed from materials selected from the group consisting of metals, carbon,
silicon, germanium, polymers and composites thereof.

20 4. The microfastening system of Claim 1 wherein said nanotubes
are at least partially multi-walled.

5. The microfastening system of Claim 1 wherein the nanotubes
are functionalized to a non-linear shape.

25 6. The microfastening system of Claim 5 wherein the non-linear
nanotubes of said fastening element are selected from hooks, loops, spirals
and combinations thereof.

30 7. The microfastening system of Claim 1 wherein said nanotubes
of at least one of said fastening elements are selectively deformable.

8. The microfastening system of Claim 1 wherein said fastening elements are reusable.

5 9. A microfastener comprising:
a substrate including an attachment surface; and
a plurality of functionalized non-linear nanotubes attached to and
extending from said attachment surface.

10 10. The microfastener of Claim 9 wherein said substrate is formed
from materials selected from the group consisting of metals, carbon, silicon,
germanium, polymers and composites thereof.

15 11. The microfastener of Claim 9 wherein said nanotubes are at
least partially multi-walled.

12. The microfastener of Claim 9 wherein the non-linear nanotubes
of said fastening element are selected from hooks, loops, spirals and
combinations thereof.

20 13. The microfastener of Claim 9 wherein at least some of the
nanotubes of said microfastener are selectively deformable.

14. A method of manufacturing a microfastener comprising the steps
of:

25 a) providing a substrate having an attachment surface;
b) introducing a plurality of open ended nanotubes to said
substrate whereby said nanotubes are attracted to said attachment surface
and become affixed thereto.

30 15. The method of Claim 14 wherein said nanotubes are
functionalized prior to attaching to said substrate.

16. The method of Claim 14 wherein said nanotubes are functionalized during attachment to said substrate.

17. The method of Claim 14 wherein said nanotubes are functionalized after attachment to said substrate.

18. The method of Claim 14 wherein said substrate is formed from materials selected from the group consisting of metals, carbon, silicon, germanium, polymers and composites thereof.

19. The method of Claim 14 wherein said nanotubes are at least partially multi-walled.

20. The method of Claim 14 wherein the non-linear nanotubes of said fastening element are selected from hooks, loops, spirals and combinations thereof.

21. The method of Claim 14 wherein at least some of said nanotubes are selectively deformable.

22. The method of Claim 14 wherein said nanotubes are attached to said substrate in the presence of an electric field.

23. The method of Claim 14 wherein said microfastener is reusable.

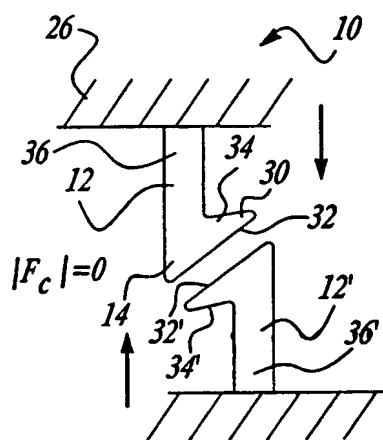


Fig-1a

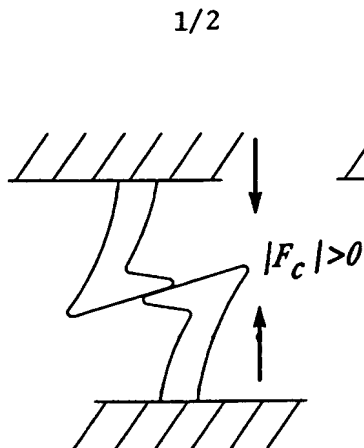


Fig-1b

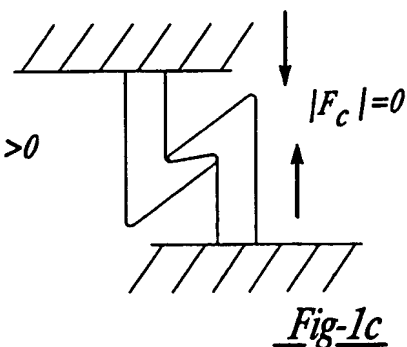


Fig-1c

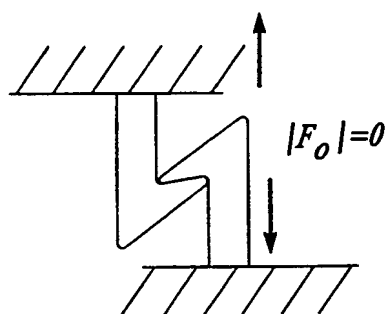


Fig-1d

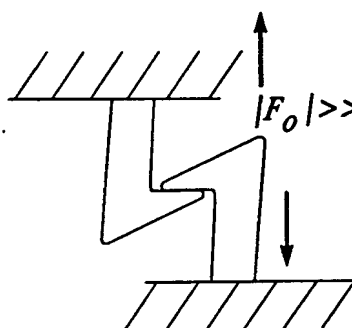


Fig-1e

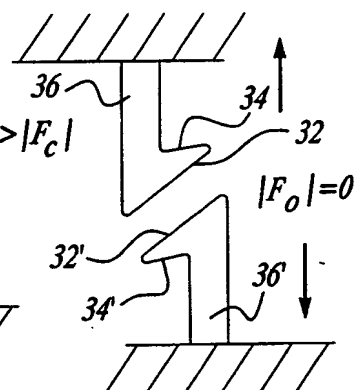


Fig-1f

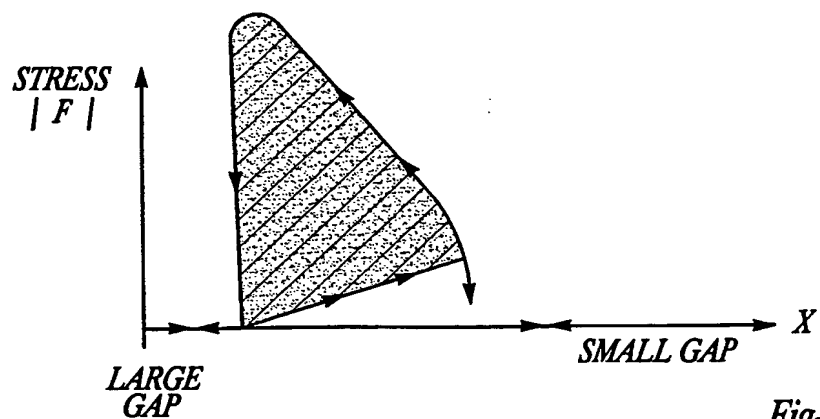


Fig-2

2/2

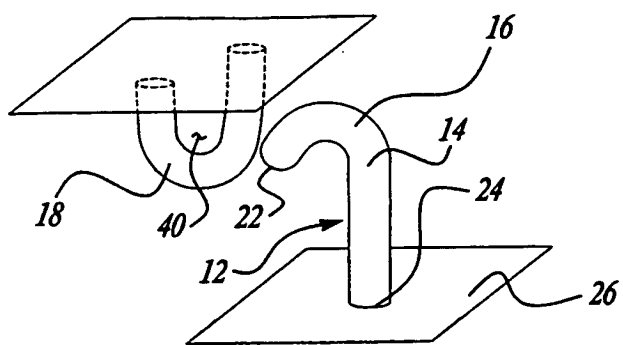


Fig-3a

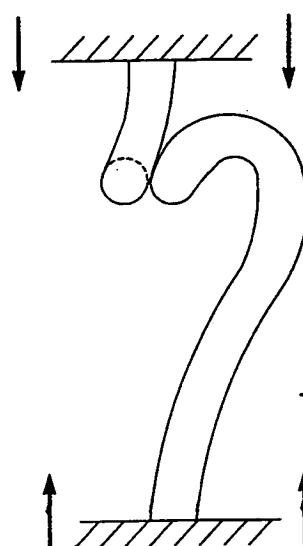


Fig-3b

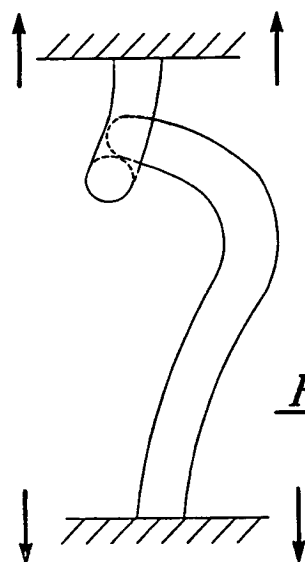


Fig-3c



Fig-3d

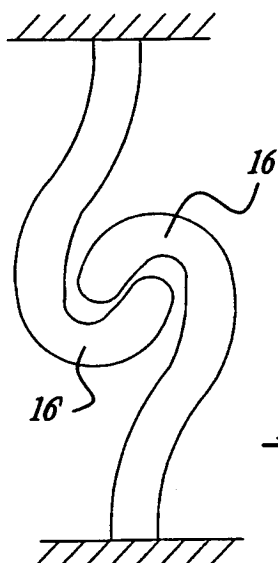


Fig-4a

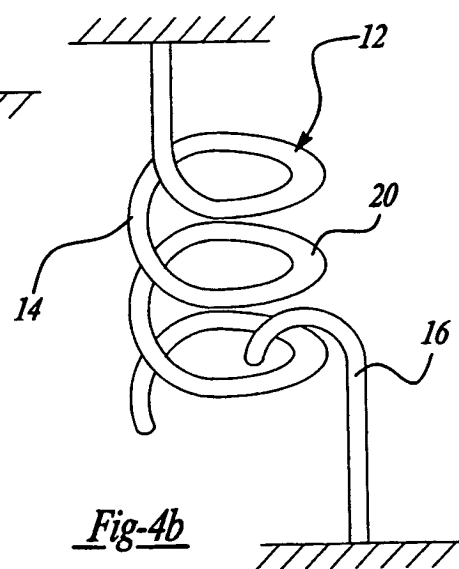


Fig-4b

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US99/02897

A. CLASSIFICATION OF SUBJECT MATTER

IPC(6) :A44B 18/00

US CL :24/442

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 24/442, 444, 446-452; 423/447.1; 428/420, 100

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

DIALOG OneSearch, Chinese Patents ABS 04/1985-02/1999, JAPIO 10/1976-11/1998, DERWENT WPI 1963-1999, US Pat.

Full 1990-1999

search terms: nanotube, microfastener, Velcro

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X -- A	US, 5,464,987 A (IHARA et al.) 07 November 1995 (07/11/95), see the key structure of figure 10.	1-6, 9-12, 14, 15 and 18-20 ----- 7, 8, 13, 16, 17 and 21-23
A	US 5,657,516 A (BERG et al.) 19 August 1997 (19/08/97), see the hollow elements of figure 6.	1
A	US 3,921,258 A (BRUMLIK) 25 November 1975 (25/11/75), see the tubular gripping member 10, col. 3, lines 12-14	1
A	US 3,889,322 A (BRUMLIK) 17 June 1975 (17/06/75), see figures 12g and 12h.	1

☒ Further documents are listed in the continuation of Box C.☐ See patent family annex.

* Special categories of cited documents:	*T* later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
A document defining the general state of the art which is not considered to be of particular relevance	*X* document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
B earlier document published on or after the international filing date	*Y* document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
L document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)	*Z* document member of the same patent family
O document referring to an oral disclosure, use, exhibition or other means	
P document published prior to the international filing date but later than the priority date claimed	

Date of the actual completion of the international search

01 JUNE 1999

Date of mailing of the international search report

17 JUN 1999

Name and mailing address of the ISA/US
Commissioner of Patents and Trademarks
Box PCT
Washington, D.C. 20231

Facsimile No. (703) 305-3230

Authorized officer

JAMES R. BRITTAIN

Telephone No. (703) 308-2168

INTERNATIONAL SEARCH REPORT

International application No.

PCT/US99/02897

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 4,531,733 A (HALL) 30 June 1985 (30/06/85), see the hollow prongs 206, col. 6, lines 63-64.	1

PATENT COOPERATION TREATY

PCT

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)



Applicant's or agent's file reference 655000017POA	FOR FURTHER ACTION See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416)	
International application No. PCT/US99/02897	International filing date (day/month/year) 11 FEBRUARY 1999	Priority date (day/month/year) 12 FEBRUARY 1998
International Patent Classification (IPC) or national classification and IPC IPC(7): A44B 18/00 and US Cl.: 24/442		
Applicant BOARD OF TRUSTEES OPERATING MICHIGAN STATE UNIVERSITY		

1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.

2. This REPORT consists of a total of 3 sheets.

☐ This report is also accompanied by ANNEXES, i.e., sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority. (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).

These annexes consist of a total of 0 sheets.

3. This report contains indications relating to the following items:

- I ☒ Basis of the report
- II ☐ Priority
- III ☐ Non-establishment of report with regard to novelty, inventive step or industrial applicability
- IV ☐ Lack of unity of invention
- V ☒ Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
- VI ☐ Certain documents cited
- VII ☐ Certain defects in the international application
- VIII ☐ Certain observations on the international application

Date of submission of the demand 02 SEPTEMBER 1999	Date of completion of this report 23 MAY 2000
Name and mailing address of the IPEA/US Commissioner of Patents and Trademarks Box PCT Washington, D.C. 20231	Authorized officer <i>James R. Brittain</i> JAMES R. BRITTAİN
Facsimile No. (703) 305-3230	Telephone No. (703) 308-2168

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No.

PCT/US99/02897

I. Basis of the report**1. With regard to the elements of the international application:***☒ the international application as originally filed☒ the description:

pages 1-7

pages NONE, as originally filed

pages NONE, filed with the demand

pages NONE, filed with the letter of

☒ the claims:

pages 8-10

pages NONE, as originally filed

pages NONE, as amended (together with any statement) under Article 19

pages NONE, filed with the demand

pages NONE, filed with the letter of

☒ the drawings:

pages 1-2

pages NONE, as originally filed

pages NONE, filed with the demand

pages NONE, filed with the letter of

☒ the sequence listing part of the description:

pages NONE

pages NONE, as originally filed

pages NONE, filed with the demand

pages NONE, filed with the letter of

2. With regard to the language, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.

These elements were available or furnished to this Authority in the following language _____ which is:

☐ the language of a translation furnished for the purposes of international search (under Rule 23.1(b)).☐ the language of publication of the international application (under Rule 48.3(b)).☐ the language of the translation furnished for the purposes of international preliminary examination (under Rules 55.2 and/or 55.3).**3. With regard to any nucleotide and/or amino acid sequence disclosed in the international application, the international preliminary examination was carried out on the basis of the sequence listing:**☐ contained in the international application in printed form.☐ filed together with the international application in computer readable form.☐ furnished subsequently to this Authority in written form.☐ furnished subsequently to this Authority in computer readable form.☐ The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.☐ The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.**4. ☒ The amendments have resulted in the cancellation of:**☒ the description, pages NONE☒ the claims, Nos. NONE☒ the drawings, sheets/fig NONE**5. ☐ This report has been drawn as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed, as indicated in the Supplemental Box (Rule 70.2(c)).****

* Replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to this report since they do not contain amendments (Rules 70.16 and 70.17).

**Any replacement sheet containing such amendments must be referred to under item 1 and annexed to this report.

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No.

PCT/US99/02897

V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement**1. statement**

Novelty (N)	Claims	<u>7, 8, 13, 16, 17, and 21-23</u>	YES
	Claims	<u>1-6, 9-12, 14, 15, and 18-20</u>	NO
Inventive Step (IS)	Claims	<u>7, 8, 13, 16, 17, and 21-23</u>	YES
	Claims	<u>1-6, 9-12, 14, 15, and 18-20</u>	NO
Industrial Applicability (IA)	Claims	<u>1-23</u>	YES
	Claims	<u>NONE</u>	NO

2. citations and explanations (Rule 70.7)

Claims 1-6, 9-12, 14, 15, and 18-20 lack novelty under PCT Article 33(2) as being anticipated by Ihara et al. (US 5,464,987). Ihara et al. discloses in the key structure of figure 10 a first fastening element (in the center of the page) including a plurality of extending nanotubes and a second fastening element including a plurality of extending nanotubes whereby the extending nanotubes of each fastening element are mechanically interconnected. As to claim 4, multi-walled nanotubes are inherent structures.

Claims 7, 8, 13, 16, 17, and 21-23 meet the criteria set out in PCT Article 33(2)-(4), because the prior art does not teach or fairly suggest the modification of the interengaging fastening system of Ihara et al. wherein the nanotubes are selectively deformable or are attaching the nanotubes in the presence of an electric field.

----- NEW CITATIONS -----
NONE

09/601540

REC'D 28 NOV 2000

15

PATENT COOPERATION TREATY

WIPO

PCT

PCT

RECEIVED

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

FEB 14 2001

(PCT Article 36 and Rule 70)

TO 3600 MAIL ROOM

Applicant's or agent's file reference 655000017POA	FOR FURTHER ACTION See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416)	
International application No. PCT/US99/02897	International filing date (day/month/year) 11 FEBRUARY 1999	Priority date (day/month/year) 12 FEBRUARY 1998
International Patent Classification (IPC) or national classification and IPC IPC(6): A44B 18/00 and US Cl.: 24/442		
Applicant BOARD OF TRUSTEES OPERATING MICHIGAN STATE UNIVERSITY		

1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.

2. This REPORT consists of a total of 4 sheets.

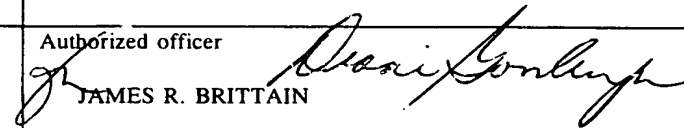
☒ This report is also accompanied by ANNEXES, i.e., sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority. (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).

These annexes consist of a total of 8 sheets.

3. This report contains indications relating to the following items:

- I ☒ Basis of the report
- II ☐ Priority
- III ☐ Non-establishment of report with regard to novelty, inventive step or industrial applicability
- IV ☐ Lack of unity of invention
- V ☒ Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
- VI ☐ Certain documents cited
- VII ☐ Certain defects in the international application
- VIII ☐ Certain observations on the international application

**CORRECTED
VERSION**

Date of submission of the demand 02 SEPTEMBER 1999	Date of completion of this report 23 MAY 2000
Name and mailing address of the IPEA/US Commissioner of Patents and Trademarks Box PCT Washington, D.C. 20231	Authorized officer  JAMES R. BRITTAI
Facsimile No. (703) 305-3230	Telephone No. (703) 308-2168

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No.

PCT/US99/02897

I. Basis of the report

1. With regard to the **elements** of the international application:*

- ☐ the international application as originally filed
- ☒ the description:
pages _____ (See Attached) _____, as originally filed
pages _____, filed with the demand
pages _____, filed with the letter of _____
- ☒ the claims:
pages _____ (See Attached) _____, as originally filed
pages _____, as amended (together with any statement) under Article 19
pages _____, filed with the demand
pages _____, filed with the letter of _____
- ☒ the drawings:
pages _____ (See Attached) _____, as originally filed
pages _____, filed with the demand
pages _____, filed with the letter of _____
- ☒ the sequence listing part of the description:
pages _____ (See Attached) _____, as originally filed
pages _____, filed with the demand
pages _____, filed with the letter of _____

2. With regard to the **language**, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.
These elements were available or furnished to this Authority in the following language _____ which is:

- ☐ the language of a translation furnished for the purposes of international search (under Rule 23.1(b)).
- ☐ the language of publication of the international application (under Rule 48.3(b)).
- ☐ the language of the translation furnished for the purposes of international preliminary examination (under Rules 55.2 and/or 55.3).

3. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international preliminary examination was carried out on the basis of the sequence listing:

- ☐ contained in the international application in printed form.
- ☐ filed together with the international application in computer readable form.
- ☐ furnished subsequently to this Authority in written form.
- ☐ furnished subsequently to this Authority in computer readable form.
- ☐ The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.
- ☐ The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.

4. ☒ The amendments have resulted in the cancellation of:

- ☒ the description, pages _____ NONE _____
- ☒ the claims, Nos. _____ NONE _____
- ☒ the drawings, sheets/fig _____ NONE _____

5. ☐ This report has been drawn as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed, as indicated in the Supplemental Box (Rule 70.2(c)).**

* Replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to this report since they do not contain amendments (Rules 70.16 and 70.17).

**Any replacement sheet containing such amendments must be referred to under item 1 and annexed to this report.

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No.

PCT/US99/02897

V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. statement

Novelty (N)	Claims <u>1-43</u>	YES
	Claims <u>NONE</u>	NO
Inventive Step (IS)	Claims <u>1-43</u>	YES
	Claims <u>NONE</u>	NO
Industrial Applicability (IA)	Claims <u>1-43</u>	YES
	Claims <u>NONE</u>	NO

2. citations and explanations (Rule 70.7)

Claims 1-43 meet the criteria set out in PCT Article 33(2)-(4), because the prior art does not teach or fairly suggest the modification of the interengaging fastening system of Ihara et al. wherein the nanotubes are selectively deformable, are hooks, spirals or have a free standing end which is free of the attachment surface.

----- NEW CITATIONS -----
NONE

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No.

PCT/US99/02897

Supplemental Box

(To be used when the space in any of the preceding boxes is not sufficient)

Continuation of: Boxes I - VIII

Sheet 10

I. BASIS OF REPORT:

This report has been drawn on the basis of the description,
page(s) 1-7, as originally filed.
page(s) NONE, filed with the demand.
and additional amendments:
NONE

This report has been drawn on the basis of the claims,
page(s) NONE, as originally filed.
page(s) NONE, as amended under Article 19.
page(s) NONE, filed with the demand.
and additional amendments:
Pages 8-15, filed with the letter of 27 October 2000.

This report has been drawn on the basis of the drawings,
page(s) 1-2, as originally filed.
page(s) NONE, filed with the demand.
and additional amendments:
NONE

This report has been drawn on the basis of the sequence listing part of the description:
page(s) NONE, as originally filed.
pages(s) NONE, filed with the demand.
and additional amendments:
NONE

CLAIMS

1. A microfastening system comprising:
a first fastening element including a plurality of extending nanotubes; and
a second fastening element including a plurality of extending nanotubes, at least some of which comprise nanotubes selected from the group consisting of
 - a) hooks, and
 - b) spirals,whereby upon joining said first and second fastening elements, the extending nanotubes from each element become mechanically interconnected.
2. The microfastening system of Claim 1 wherein said at least one of first and second fastening elements further comprise a substrate from which said nanotubes extend.
3. The microfastening system of Claim 2 wherein said substrate is formed from materials selected from the group consisting of metals, carbon, silicon, germanium, polymers and composites thereof.
4. The microfastening system of Claim 1 wherein said nanotubes are at least partially multi-walled.

5. The microfastening system of Claim 1 wherein the nanotubes are functionalized to a non-linear shape.
6. The microfastening system of Claim 5 wherein the non-linear nanotubes of said first fastening element are selected from hooks, loops, spirals and combinations thereof.
7. The microfastening system of Claim 1 wherein said nanotubes of at least one of said fastening elements are selectively deformable.
8. The microfastening system of Claim 1 wherein said fastening elements are reusable.
9. A microfastener comprising:
 - a substrate including an attachment surface; and
 - a plurality of functionalized non-linear nanotubes attached to and extending from said attachment surface, wherein the nanotubes have a free standing end which is free of the surface.
10. The microfastener of Claim 9 wherein said substrate is formed from materials selected from the group consisting of metals, carbon, silicon, germanium, polymers and composites thereof.

11. The microfastener of Claim 9 wherein said nanotubes are at least partially multi-walled.
12. The microfastener of Claim 9 wherein the non-linear nanotubes of said fastening element further comprise loops.
13. The microfastener of Claim 9 wherein at least some of the nanotubes of said microfastener are selectively deformable.
14. A method of manufacturing a microfastener having nanotubes with two ends, comprising the steps of:
- a) providing a substrate having an attachment surface;
 - b) introducing a plurality of open ended nanotubes to said substrate whereby said nanotubes are attracted to said attachment surface and become affixed thereto, wherein at least some of the nanotubes become affixed at only one end.
15. The method of Claim 14 wherein said nanotubes are functionalized prior to attaching to said substrate.
16. The method of Claim 14 wherein said nanotubes are functionalized during attachment to said substrate.

17. The method of Claim 14 wherein said nanotubes are functionalized after attachment to said substrate.

18. The method of Claim 14 wherein said substrate is formed from materials selected from the group consisting of metals, carbon, silicon, germanium, polymers and composites thereof.

19. The method of Claim 14 wherein said nanotubes are at least partially multi-walled.

20. The method of Claim 14 wherein the non-linear nanotubes of said fastening element further comprise loops.

21. The method of Claim 14 wherein at least some of said nanotubes are selectively deformable.

22. The method of Claim 14 wherein said nanotubes are attached to said substrate in the presence of an electric field.

23. The method of Claim 14 wherein said microfastener is reusable.

24. The microfastening system comprising:
a first fastening element including a plurality of extending nanotubes; and
a second fastening element including a plurality of extending nanotubes, wherein said nanotubes of at least one of said fastening elements are selectively deformable;
whereby upon joining said first and second fastening elements, the extending nanotubes from each element become mechanically interconnected.
25. The microfastening system of Claim 24 wherein said at least one of first and second fastening elements further comprise a substrate from which said nanotubes extend.
26. The microfastening system of Claim 25 wherein said substrate is formed from materials selected from the group consisting of metals, carbon, silicon, germanium, polymers and composites thereof.
27. The microfastening system of Claim 24 wherein said nanotubes are at least partially multi-walled.
28. The microfastening system of Claim 24 wherein the nanotubes are functionalized to a non-linear shape.

29. The microfastening system of Claim 28 wherein the non-linear nanotubes of said fastening element are selected from hooks, loops, spirals and combinations thereof.

30. The microfastening system of Claim 24 wherein said fastening elements are reusable.

31. A microfastener comprising:
a substrate including an attachment surface; and
a plurality of functionalized selectively deformable non-linear nanotubes attached to and extending from said attachment surface.

32. The microfastener of Claim 31 wherein said substrate is formed from materials selected from the group consisting of metals, carbon, silicon, germanium, polymers and composites thereof.

33. The microfastener of Claim 31 wherein said nanotubes are at least partially multi-walled.

34. The microfastener of Claim 31 wherein the non-linear nanotubes of said fastening element are selected from hooks, loops, spirals and combinations thereof.

35. A method of manufacturing a microfastener comprising the steps of:
- a) providing a substrate having an attachment surface;
 - b) introducing a plurality of open ended selectively deformable non-linear nanotubes to said substrate whereby said nanotubes are attracted to said attachment surface and become affixed thereto.
36. The method of Claim 35 wherein said nanotubes are functionalized prior to attaching to said substrate.
37. The method of Claim 35 wherein said nanotubes are functionalized during attachment to said substrate.
38. The method of Claim 35 wherein said nanotubes are functionalized after attachment to said substrate.
39. The method of Claim 35 wherein said substrate is formed from materials selected from the group consisting of metals, carbon, silicon, germanium, polymers and composites thereof.
40. The method of Claim 35 wherein said nanotubes are at least partially multi-walled.

41. The method of Claim 35 wherein the non-linear nanotubes of said microfastener are selected from hooks, loops, spirals and combinations thereof.

42. The method of Claim 35 wherein said nanotubes are attached to said substrate in the presence of an electric field.

43. The method of Claim 35 wherein said microfastener is reusable.

2907169